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Standard Method of Test for
SAPONIFICATION VALUE OF DRYING OILS, FATTY ACIDS, AND POLYMERIZED FATTY ACIDS[1]

This Standard is issued under the fixed designation D 1962; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers the determination of the saponification value of drying oils, bodied oils, fatty acids, and polymerized fatty acids.

2. Definition

2.1 *saponification value*—a measure of the alkali reactive groups in oils and fatty acids and is expressed as the number of milligrams of potassium hydroxide that react with 1 g of sample.

3. Apparatus

3.1 *Erlenmeyer Flasks*, wide-mouth, alkali-resistant, 250 or 300-ml capacity.

3.2 *Condenser Loop.*

NOTE 1—Suitable condenser loops are shown in Figs. 1 and 2 of ASTM Method D 305, Test for Acetone Extract in Black Pigments.[2]

3.3 *Steam Bath.*

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform the the Specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to ASTM Specification D 1193, for Reagent Water.[2]

4.3 *Phenolphthalein Indicator Solution*—Dissolve 1 g of phenolphthalein in 100 ml of ethyl alcohol (95%) or denatured alcohol conforming to Formula No. 30 or 3A of the U. S. Bureau of Internal Revenue.

NOTE 2—A "masked phenolphthalein indicator" may be used with off-color materials. Prepare by dissolving 1.6 g of phenolphthalein and 2.7 g of methylene blue in 500 ml of denatured alcohol. Adjust the pH with sodium hydroxide (NaOH) or KOH solution so that the greenish blue color is faintly tinged with purple. The color change is from green to purple when going from acid to alkali.

4.4 *Potassium Hydroxide, Alcoholic Solution*—Place 5 to 10 g of potassium hydroxide (KOH) in a 2-litre flask and add 1 to 1.5 litres of ethyl alcohol (95 percent) or denatured alcohol conforming to Formula No. 30 or 3A of the U. S. Bureau of Internal Revenue. Boil on a water bath under a reflux condenser for 30 to 60 min. Distill and collect the alcohol. Dissolve 40 g of KOH in 1 litre of the distilled alcohol, keeping the temperature below 15.5 C while the alkali is being dissolved. This solution should remain clear.

4.5 *Sulfuric or Hydrochloric Acid, Standard* (*0.5 N*)—Add about 15 ml of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) or 45 ml of concentrated hydrochloric acid (HCl, sp gr 1.19) to about 900 ml of water, cool, and dilute to 1 litre. Standardize against freshly standardized sodium hydroxide (NaOH) solution or by any other accurate method.

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[1] This method is under the jurisdiction of ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

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[2] *1974 Annual Book of ASTM Standards*, Part 29.

[3] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D. C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N. Y., and the "United States Pharmacopeia."

5. Procedure

5.1 To an Erlenmeyer flask, transfer a sample weight of such size, weighed to the nearest 0.001 g, that the back titration is 45 to 55% of the blank. Add 25 ml of alcoholic KOH solution to the sample and to one or more additional flasks to be carried through as blanks. Place a condenser loop inside the neck of each flask and heat on the steam bath for 1 h.

NOTE 3—Certain synthetic oils are not completely saponified in 1 h. Run samples of chemically modified drying oils in duplicate, using 1 and 2-h heating periods to establish completeness of saponification. If the 2-h heating gives appreciably higher results than the 1-h run, additional determinations using 4 and 6-h heating periods should be run to establish the time required for complete reaction.

5.2 Cool the solution, add phenolphthalein indicator (Note 2), and titrate with 0.5 N H_2SO_4 or HCl until the pink color has just disappeared.

6. Calculation

6.1 Calculate the saponification number as follows:

$$\text{Saponification number} = [(B - A)N \times 56.1]/C$$

where:

A = millilitres of H_2SO_4 or HCl required for titration of the sample,
B = millilitres of H_2SO_4 or HCl required for titration of the blank,
N = normality of the H_2SO_4 or HCl, and
C = grams of sample used.

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